6.1.2 Preparing and purifying organic solids

Name: _____ Class: _____ Date: _____ **Total: 19 marks**

KEY WORDS recrystallisation • suction filtration • fume cupboard

Objective

Prepare and purify an organic solid the way the scheme credits: safety matched to each hazard, showing which reagent's volume may be inexact, washing away soluble impurities, recrystallising below the melting temperature, and certifying purity by melting temperature. Original practice for WCH16's synthesis demands (Summer 2025 Q4; CP16 context).

1 Key knowledge and worked examples

■ Safety named to the hazard

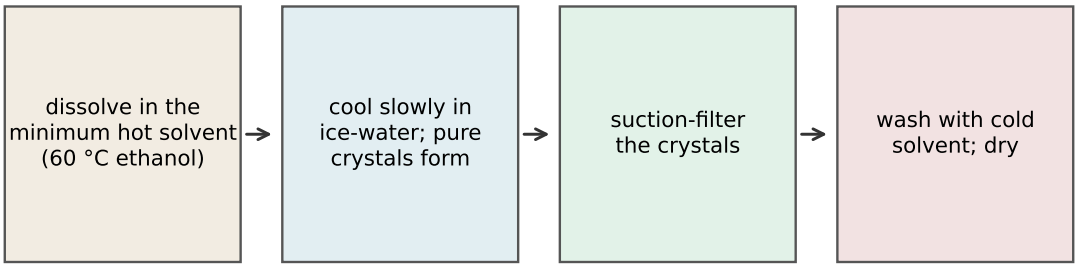
Phenol is toxic by skin absorption → **gloves** when weighing. Benzoyl chloride is toxic by inhalation and corrosive → **fume cupboard**, and the stopper is released every few minutes (pressure). Corrosive + flammable solvents → no flames.

■ Why a volume need not be exact

Show it by calculation. Phenol: $5.00/94.0 = 0.0532$ mol. Benzoyl chloride: $9\ \text{cm}^3 \times 1.21\ \text{g cm}^{-3} = 10.89\ \text{g} = 10.89/140.5 = 0.0775$ mol. The chloride is in **excess**, so its exact volume cannot change the amount of product – phenol limits the yield.

■ Washing and recrystallising

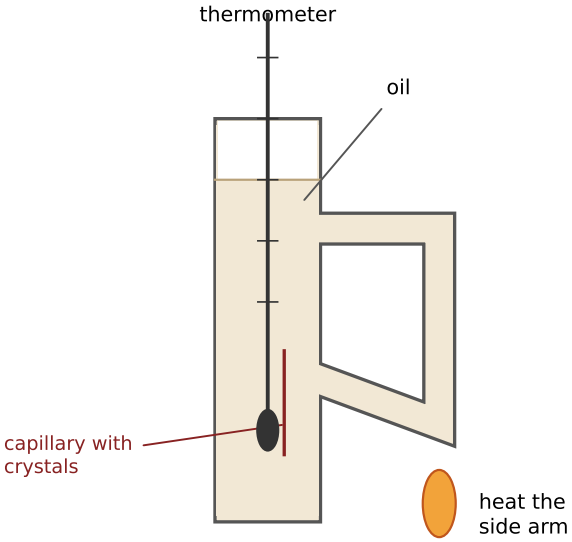
Water wash removes **soluble** impurities: excess benzoyl chloride, HCl, NaOH, NaCl. "Insoluble impurities" scores zero. Recrystallisation: dissolve in the minimum hot solvent at **60 °C – below ethanol's boiling point and below the product's 71 °C melting temperature, so the crystals do not melt**; cool slowly in ice-water; suction-filter; wash with **cold** solvent – impurities leave, crystals stay.



impurities stay dissolved at every stage; the product is only soluble hot

Purity by melting temperature

A sharp melt at the data-book value (phenyl benzoate 71 °C) means pure; depressed and broad means impure. Apparatus – Thiele tube (or boiling tube) of oil, labelled thermometer in the liquid, capillary of crystals, heated by a **flame** (the question bans electrical equipment).



2 Practice

2.1 State the safety precaution when weighing phenol, and the reason it is that precaution rather than goggles. [2]

2.2 Give the reason the reaction vessel is stoppered and shaken with careful pressure release. [1]

2.3 The crude product is washed with water. Give two substances the wash removes. [2]

2.4 Why is the crude solid dissolved at 60 °C instead of letting the ethanol boil? [1]

3 Exam-style questions

3.1 Benzoyl chloride (density 1.21 g cm^{-3}, M_r 140.5) is added to 5.00 g of phenol (M_r 94.0). Show by calculation that the volume of benzoyl chloride does not need to be measured exactly. [4]

3.2 Describe how the crude product is recrystallised and dried, giving the reason for the temperature of dissolution and for washing the crystals with cold rather than warm ethanol. [6]

3.3 Draw and label the apparatus used to measure the melting temperature of the dry crystals, assuming no electrical equipment is available. [3]